Tuning the Vacuum Distiller Optimizing Analyte Response and Chromatography

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Why Tune?

- Ensure analytes are distilled and pass through the condenser
- Chromatography can seriously degrade for some analytes if too much water or methanol is introduced to the GC column
- GC/MS apparatus, injector, and column all have differing sensitivities to water and methanol that must be accommodated for best results

First Step..Build Vdist Method

- Set vacuum distillation time to 7.5 minutes (Menu->Method->Run Method)
- Set condenser cool temperature to 30 (note: the true temperature of the condenser is very likely different from the measured temperature)
- Set other method variables as in the following slides
- After verifying all variables are correct, save method as default.m and load into vacuum distiller memory by pressing Send and Implement

Default.m

- Pre-distillation evacuation time: 0.00 minutes (min)
- Vacuum distillation time: 7.5 min
- Transfer time (cryotrap to GC): 5 min
- Condenser temperature settings
  - Heating: 95 °C
  - Cooling: 30 °C

Default.m Part2

- Cryotrap settings
  - Cryotrapping: -150 °C
  - Desorb delay time: 0 min
  - Desorb temp: 120 °C
  - Bakeout temp: 200 °C
- Transfer line (VDU to GC) temp: 200 °C
- Cryotrap bakeout and condenser purge: 7.0 min

Default.m Part3

- Flushing cycle
  - Pressurization time: 0.05 min
  - Evacuation time: 1.2 min
  - Number of cycles: 16

Stabilization times (Temperature)
  - Condenser time: 0.1 min
  - Cryotrap time: 0.3 min
- Vacuum distiller internal line temp: 95 °C
- Multiport Valve temp: 200 °C
- Autosampler lines temp: 95 °C

Step 2. Optimize Default Method to Distill ~0.3g

- Add 5 mL water (weigh) to sample vessel and attach to Port 1 (see Sample Preparation for more details)
- Perform vacuum distillation as single distillation (Menu->Run->Status-> Run) or through the Sequence Procedure (See Running Samples)
- Not necessary to run GC/MS for this step

Distillation is Complete

- If a GC/MS run is not desired abort vacuum distillation run when distillation is complete (and waiting for GC Ready) Menu->Run->Status-> Stop
- Weigh sample in container and determine water distilled
- Repeat distillation with condenser cool temperature setting lower by 10
- Continue lowering condenser cool setting until the water loss in the sample ~0.3g
- The condenser cool setting for future distillations will be that setting where ~0.3 g is distilled

Step 3.Setting the Cryotrap Desorb Temperature and the "to GC" Transfer Time

- Setting the cryotrap desorb temperature and "to GC" transfer times are critical to good analyte identification and integration
- Analyte resolution and peak shape are easily degraded if too much water or methanol are loaded on column.
- Injector type, column, and GC/MS sensitivity impact the optimal vacuum distillation method settings

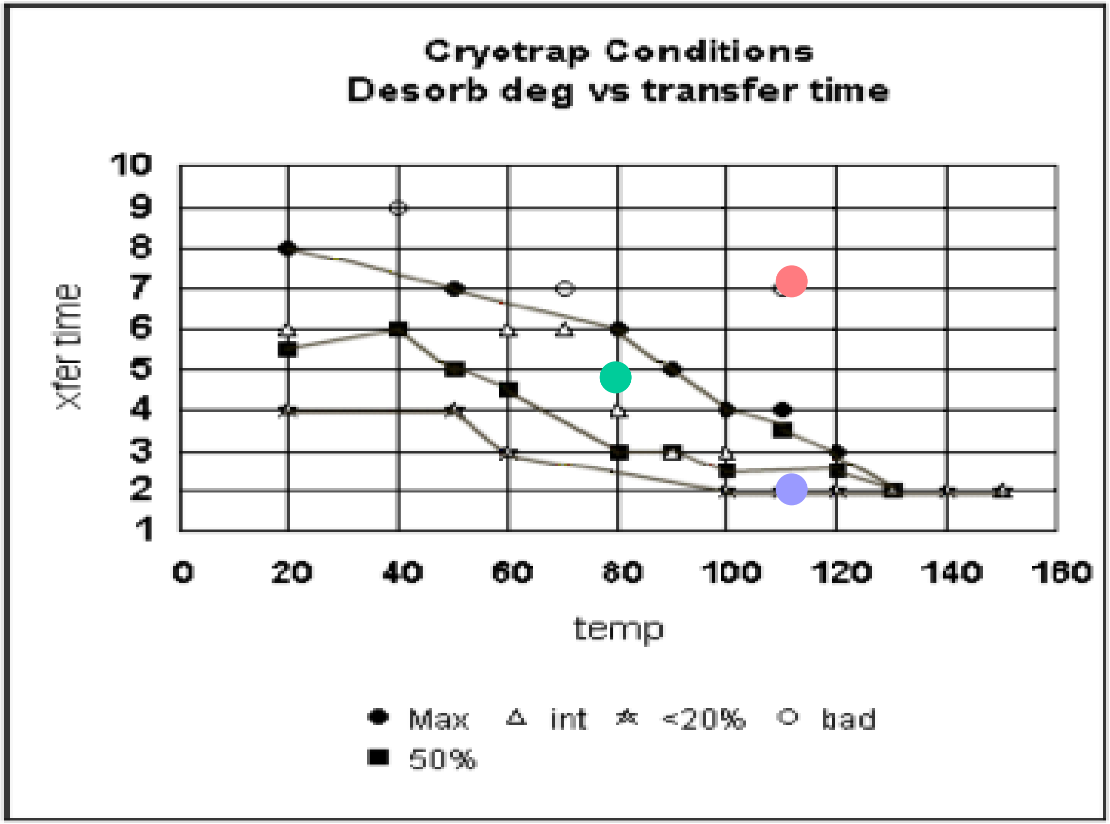
The "to GC" Transfer Time and the Cryotrap Desorb Temperature are Related

- Too much water or methanol being transferred from the cryotrap to the GC degrades chromatography
- Too much water or methanol can be the result of too long of a "to GC" transfer or the cryotrap is too hot during desorb
- Too cold of a desorb temperature or too quick of a "to GC" transfer time result in incomplete transfer of analyte and poor sensitivity

Experimental evaluation of Desorb Temperature and "to GC" Transfer Time

- Surrogate compounds were distilled and transferred to GC using various cryotrap conditions
- Surrogate GC/MS responses and peak shapes were recorded
- Results were graphed as a function of experimental conditions, desorb temperature and "to GC" transfer time

Desorb Temperature and Transfer Time Impact on Analtyes-Graph



- Desorb temperature or transfer time too great
- Desorb temperature and transfer time are balanced
- Desorb temperature or transfer time too low

Note:Graph generated with prototype distiller interfaced with jet separator and will be different for other systems

Desorb Temperature and Transfer Time Impact on Analtyes

- The line connecting solid circles represents conditions that give maximum response and acceptable chromatography
- The line connecting solid boxes represents conditions that give responses half those of the maximum response line
- The line connecting stars represents conditions that give responses 1/5 those of the maximum response line
- Open circles are those conditions that resulted in poor chromatography
- Open triangles are those conditions that resulted in good chromatography

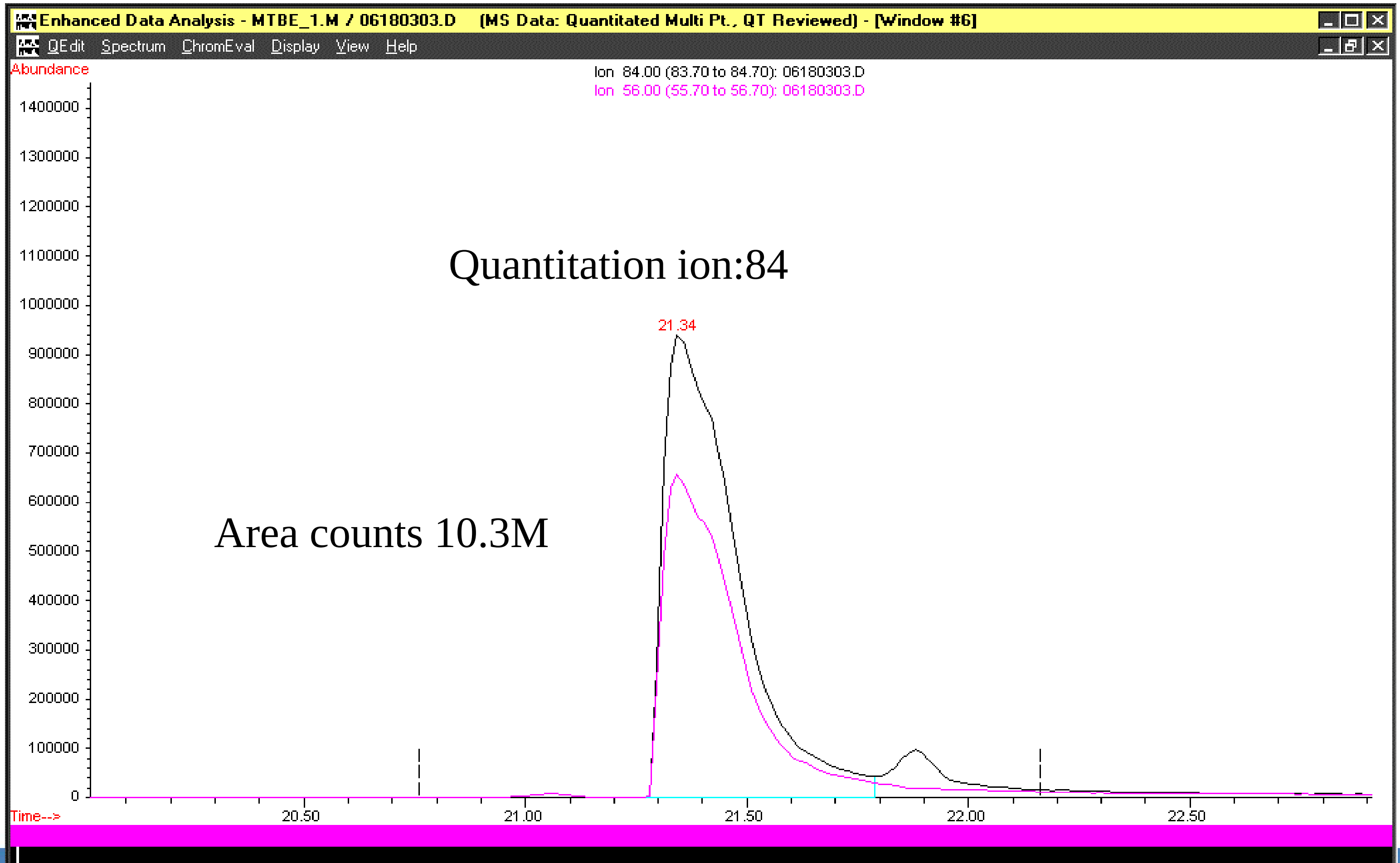
What Does the Graph Mean?

- For a large desorb temperature range there can be a “to GC” transfer time that results in good data
- For a range of transfer times there is a desorb temperature that results in good data
- Quicker “to GC” transfer times make selecting a good desorb temperature very sensitive with little room for variations
- Slower “to GC” transfer times provide a greater range of acceptable desorb temperatures

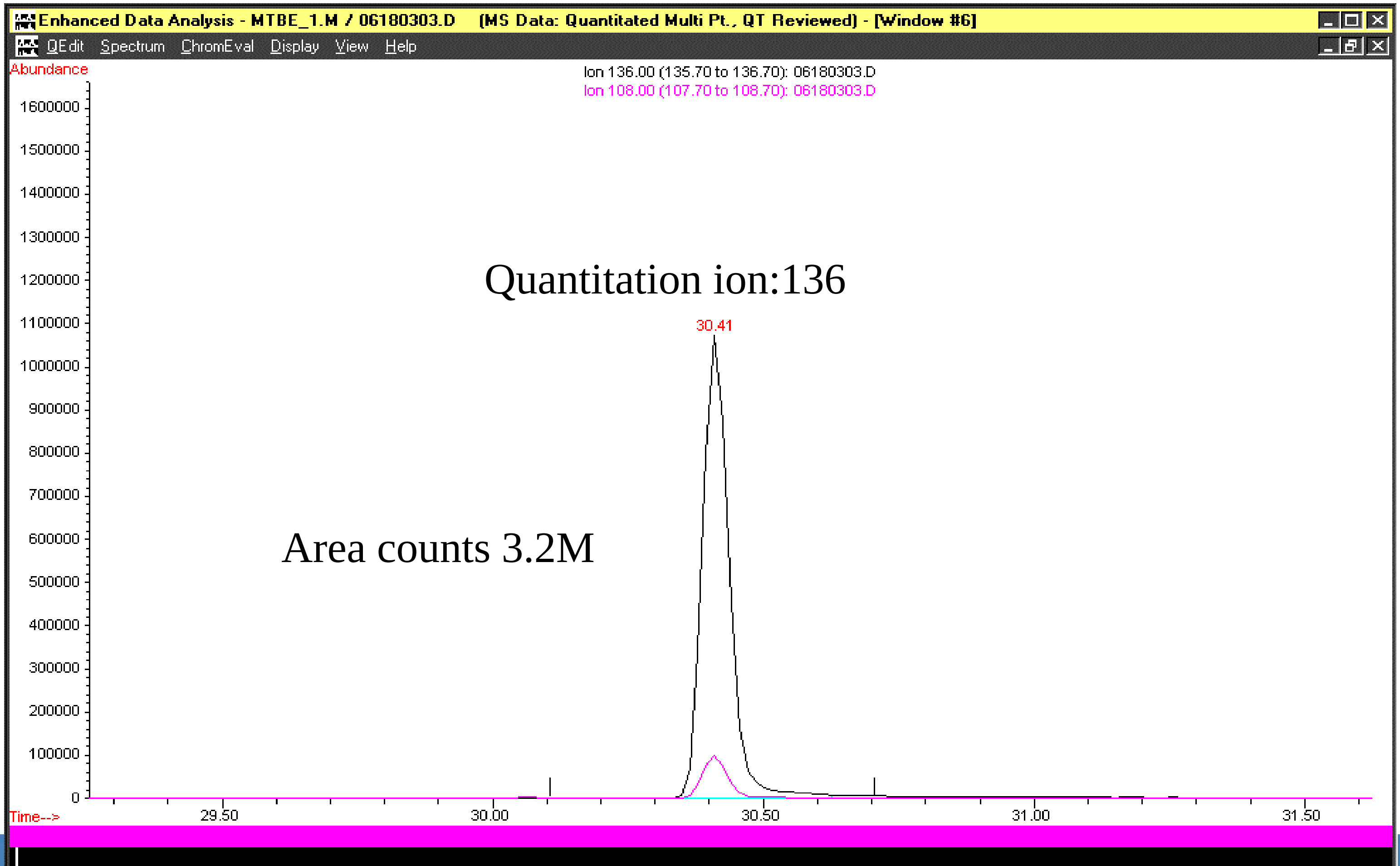
What Does the Graph Mean? Part2

- Selecting an economical “to GC” transfer time the analyst can just vary desorb temperature to “tune” the system
- The analyst should consider that >7 cryotrap volumes (~7 mL) of helium carrier gas should be passed through the trap after the cryotrap is at desorb temperature. For most circumstances a transfer time of 5 minutes is adequate
- Following slides show how “tune” severely impacts pyridine-d5 and more subtly impacts naphthalene

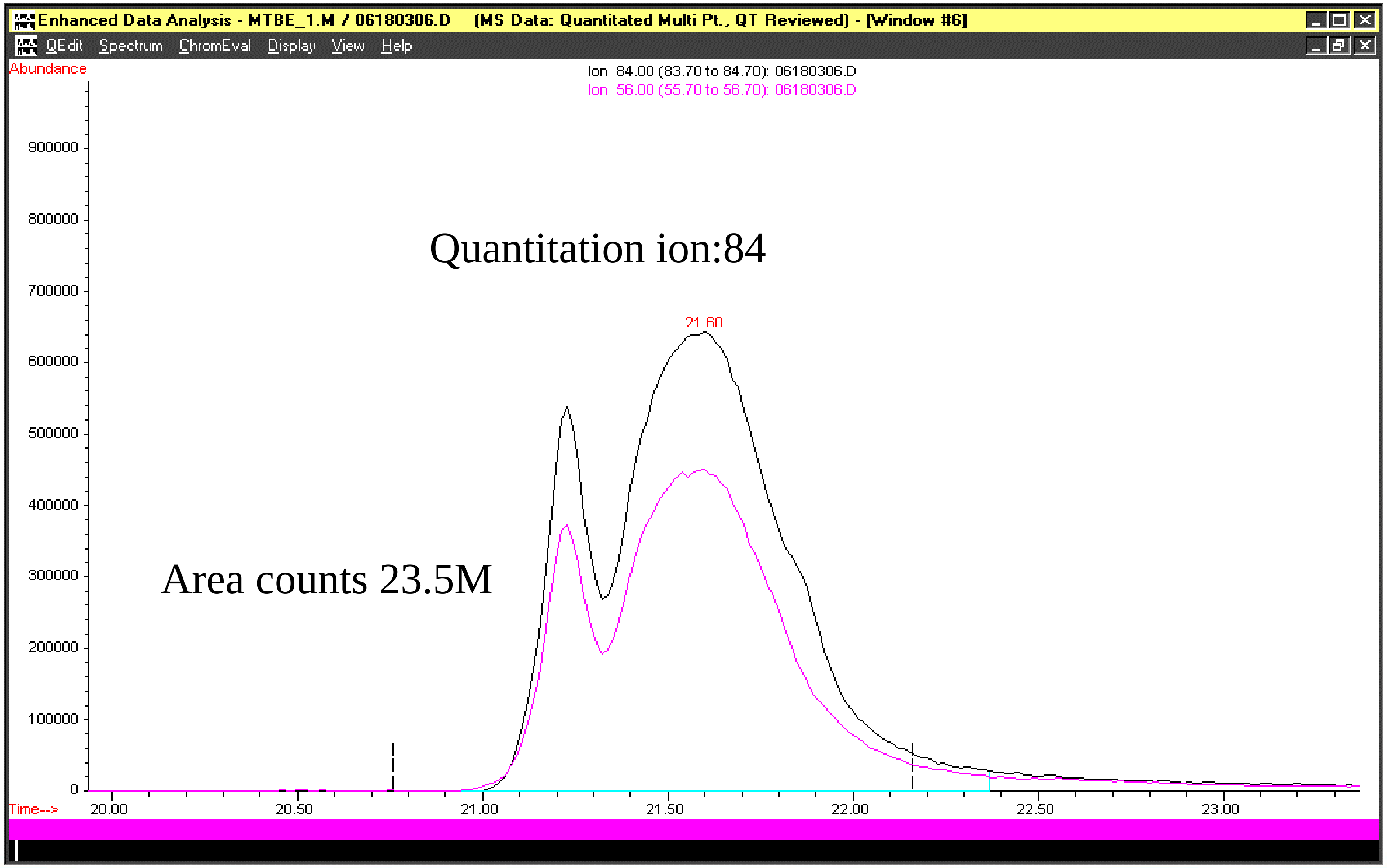
Desorb Temperature and Transfer Time Good Pyridine-d5



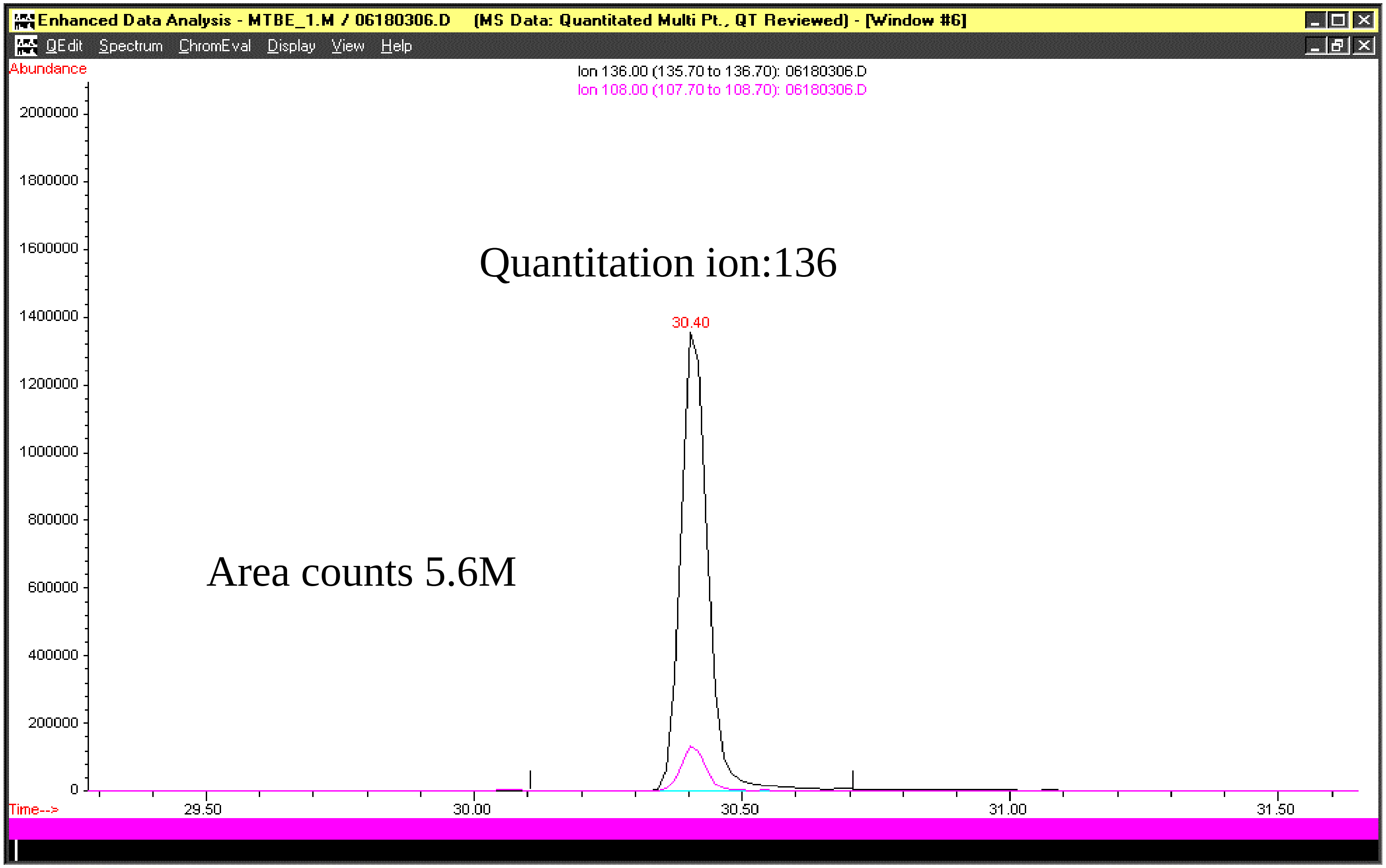
Desorb Temperature and Transfer Time Good Naphthalene-d8



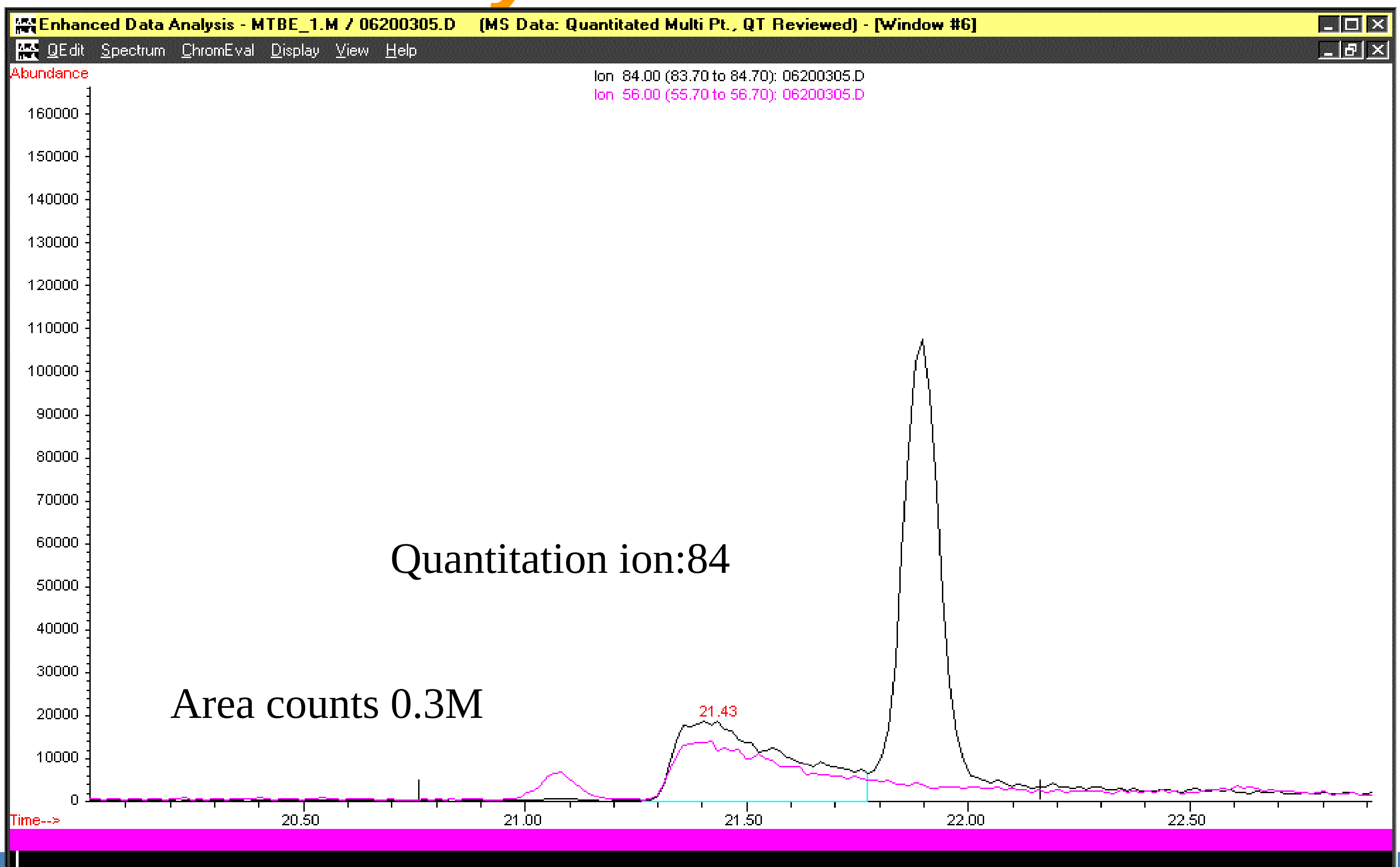
Desorb Temperature or Transfer Time too Great Pyridine-d5



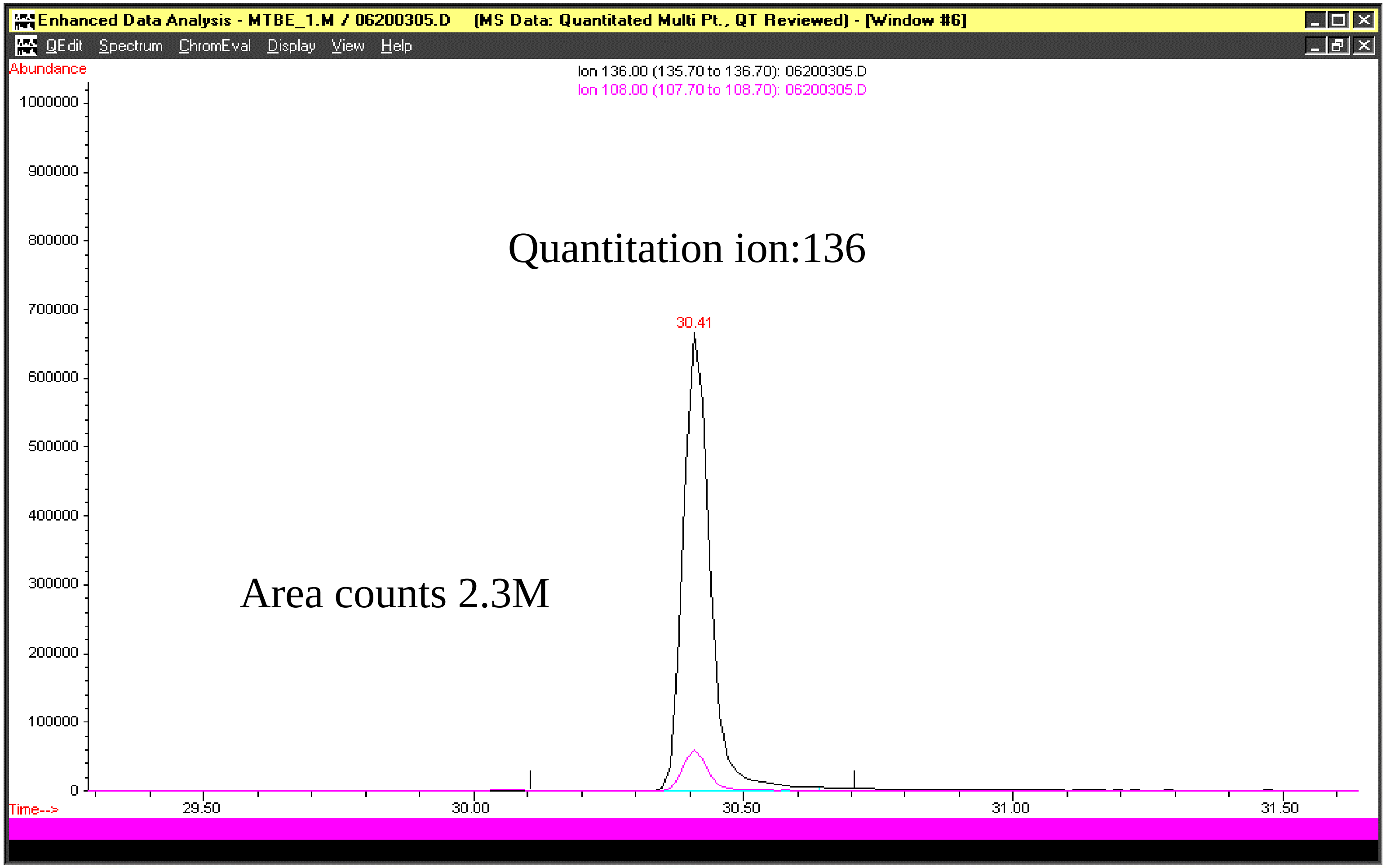
Desorb Temperature or Transfer Time too Great Naphthalene-d8



Desorb Temperature or Transfer Time too Low
Pyridine-d5



Desorb Temperature or Transfer Time too Low Naphthalene-d8



Experiments to Determine Final "Tune"

- Multiple vacuum distillations will be performed (Surrogates in 5 mL water)
- A single "to GC" transfer time will be selected and an appropriate desorbing temperature will be assigned
- A vacuum distillation method can then be created for performing method 8261 calibrations
- See the presentation "Running Samples" for performing the actual vacuum distillations

Experiment for evaluating Desorb Temperatures

- Using method used to determine the condenser cool temperature (updated for the determined condenser setting XX), set “to GC” transfer time to 5 minutes and save as a method template (example condXX.M)
- Create a method for each desorb temperatures are 140, 120, 100, 80 (example condxxcryo140.M)
- Load 4-5mL samples (containing surrogates) on the autosampler
- Set up sequence (See Running a Sequence in the Running Samples presentation) with each sample assigned one of the new methods for desorb temperatures (example condxxcryo140.M)

Experiment for Determing Desorb Temperatures..Evaluation

- Evaluate the GC/MS data generated by looking at profiles for pyridine-d5 and naphthalene-d8
- Compare the responses of naphthalene-d8 and pyridine-d5 and the peak shape of pyridine-d5
- If the "better" conditions are for 140 or 80 desorbing temperature perform an additional distillation 20 degrees more extreme
- Keeping in mind the graph displayed in slide 8, perform more distillations (5 deg increments) to determine the "good" range of desorb temperatures

Step 4. Done!

- The desorb temperature that provides good pyridine-d5 and good intensity (or mid-range of good desorb temperatures) is used to create the final method
- The system is now ready to create a calibration curve

Problems?

- If an acceptable desorb temperature is not found, the GC column may not be appropriate or the carrier gas flow may be outside normal.
- Verify ~0.3 grams of water are being distilled
- A series of transfer times can be analyzed for a single desorb temperature (120 or greater). The 5 min transfer time may not be correct for the system